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Yield Stress Reduction of DWPF Melter Feed Slurries

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The Defense Waste Processing Facility (DWPF) at the Savannah River Site vitrifies High Level Waste for repository internment. The process consists of three major steps: waste pretreatment, vitrification, and canister decontamination/sealing. The HLW consists of insoluble metal hydroxides (primarily iron, aluminum, magnesium, manganese, and uranium) and soluble sodium salts (carbonate, hydroxide, nitrite, nitrate, sulfate). The pretreatment process acidifies the sludge with nitric and formic acids, adds the glass formers as glass frit, then concentrates the resulting slurry to approximately 50 weight percent (wt%) total solids. This slurry is fed to the joule-heated melter where the remaining water is evaporated followed by calcination of the solids and conversion to glass.

The Savannah River National Laboratory (SRNL) is currently assisting DWPF efforts to increase throughput of the melter. As part of this effort, SRNL has investigated methods to increase the solids content of the melter feed to reduce the heat load required to complete the evaporation of water and allow more of the energy available to calcine and vitrify the waste. The process equipment in the facility is fixed and cannot process materials with high yield stresses, therefore increasing the solids content will require that the yield stress of the melter feed slurries be reduced.

Changing the glass former added during pretreatment from an irregularly shaped glass frit to nearly spherical beads was evaluated. The evaluation required a systems approach which included evaluations of the effectiveness of beads in reducing the melter feed yield stress as well as evaluations of the processing impacts of changing the frit morphology. Processing impacts of beads include changing the settling rate of the glass former (which effects mixing and sampling of the melter feed slurry and the frit addition equipment) as well as impacts on the melt behavior due to decreased surface area of the beads versus frit.

Beads were produced from the DWPF process frit by fire polishing. The frit was allowed to free fall through a flame, then quenched with a water spray. Approximately 90% of the frit was converted to beads by this process, as shown in Figure 1. Borosilicate beads of various diameters were also procured for initial testing.

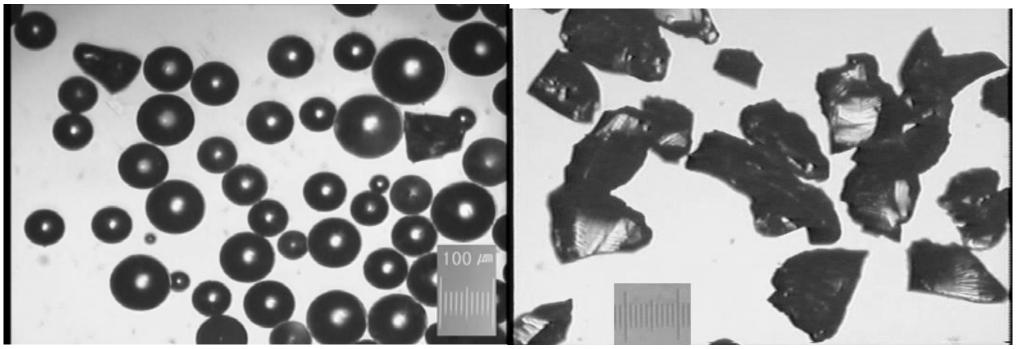

Figure 1. Beads and Unprocessed Frit 418

Yield stress reduction was measured by preparing melter feed slurries (using non-radioactive HLW simulants) that contain beads and comparing the yield stress with melter feed containing frit. The yield stress was measured with a Haake RS-150 or Haake RS-600 rheometer. The Bingham Plastic model was used to regress the data and determine the yield stress. Results of preliminary tests are shown in Table 1, with the beads resulting in a decrease of 20 to 40%[1].

Table 1. Preliminary Results: Beads versus Frit

SB2	Yield Stress	Consistency	**SB2/3**	Yield Stress	Consistency
	50 - 200 Shear Rate Up Curve Only			50 - 200 Shear Rate Up Curve Only	
Beads	Pa	cP	Beads	Pa	cP
RHEO-226-A	4.1	14.6	RHEO-229-A	2.8	16.5
RHEO-226-B	4.4	15.3	RHEO-229-B	3.0	17.3
Average	4.2	15.0	Average	2.9	16.9
Frit	Pa	cP	Frit	Pa	cP
RHEO-227-A	5.7	18.1	RHEO-228-A	4.5	20.5
RHEO-227-B	5.0	13.9	RHEO-228-B	5.5	21.6
Average	5.4	16.0	Average	5.0	21.0
% Difference	21.4	6.1	% Difference	41.5	19.7

A second set of tests was performed with beads of various diameters to determine if a decrease in diameter affected the results. Smaller particle size was shown to increase yield stress when frit is utilized. The settling rate of the beads was required to match the settling rate of the frit, therefore a decrease in particle size was anticipated. As shown in Table 2, decreasing the particle size did not reduce the effectiveness of switching from frit to beads[2].

Table 2. Impact of Frit 320 and Various Sized Beads on Rheology of SB3 SME Product

Frit/Bead (Sample ID)	Yield Stress	Consistency (Pa)	* % Reduction in Yield Stress /Consistency
Frit 320	3.26	20.53	N.A.
-140 Bead	2.64	16.05	23 / 28
-100+140 Bead	2.51	15.27	30 / 34
-70+100 Bead	2.61	12.88	25 / 59

* Relative to Frit 320 SME product

Settling tests were conducted in water, xanthan gum solutions, and in non-radioactive simulants of the HLW. The tests used time-lapse videography as well as solids sampling to evaluate the settling characteristics of beads compared to frit of the same particle size. The test with HLW simulant was performed by pouring a well-mixed 45% slurry into a 100 ml cylinder and sampling the each cylinder at the 50 ml volume for total solids as a function of time. As shown in Table 3, beads settled slightly faster than frit at a given particle size.

Table 3. Settling Tests with SB3 Melter Feed

Settling Time (Hours)	Wt% Solids of SB3/Frit 320 (-100+140 mesh)	Wt% Solids of SB3/Beads (-100+140 mesh)
2	46.52	47.17
4	45.79	48.10
6	45.67	47.36
24	45.95	49.26

Settling rate for larger frit particles (-80/+100 mesh) was much higher than the settling rate for the -100/+140 beads, so the particle size range for beads was reduced from the -80/+200 specified for frit to -100/+200 for the beads.

A preliminary melt rate evaluation was performed using a dry-fed Melt Rate Furnace (MRF) developed by SRNL. The MRF is a scoping tool designed for quick screening of melt rate. The testing consisted of preparing sufficient melter feed simulant using beads and frit to produce 500 grams of glass. This material is dried and placed into a 1200 ml stainless steel beaker. The beaker is heated from the bottom with the sides insulated for a specified time, then the amount of glass in the beaker is measured. The measured melt rates (0.45 and 0.47 in/hr) were nearly identical, but differences in the appearance of the fired beakers were noted as shown in Figure 2.

Figure 2. Top Views of the SB3/Bead 320 and SB3/Frit 320 MRF Beakers and the Cross-Sections of the Beakers

The differences in the appearances of the beakers in MRF indicate that further evaluation is necessary. Tests are planned in the Slurry-fed Melt Rate Furnace that more closely replicates the conditions in the DWPF melter.

Preliminary evaluation of the impact of beading the frit on the frit addition system were completed by conducting flow loop testing. A recirculation loop made of clear ¾" PVC piping was built with a total length of about 30 feet. The test rig had a plastic 10 gallon feed tank and a Jabsco pump. A 2" PVC bypass return loop to the feed tank was installed immediately after the pump with a ball valve to control flow in the main recirculation loop. A flow transmitter followed by both a pressure gauge and a digital pressure gauge were installed on the outlet side of the pump. A schematic of the bead pump test stand and a picture of the test stand (without the bypass line) are shown in Figures 3 and 4 respectively. Six gallon solutions of water and either -100+140 mesh beads or Frit 320 were tested. In the first test, 14 kg of water was first added to the feed tank, and then 14.0 kg of beads (to make a 50 weight percent solids solution) was slowly added with the agitator running. A minimum agitator speed was then determined. After this, the pump was started with bypass valve closed for maximum recirculation flow. Pump power, flow rate, outlet pressure, and observations of the flow in the horizontal upper section of the loop were noted. The recirculation flow was then gradually reduced and the above items recorded until settling was noted in the recirculation line. After this, the test was repeated by adding 3.11 kg of beads to make a 55 weight percent mixture. Finally, a 60 weight percent mixture was tested by adding another 3.89 kg of beads to the feed tank. This same process was planned for Frit 320, but only the initial 50 weight percent mixture could be tested before the pump impeller failed due to the erosive Frit 320 particles.

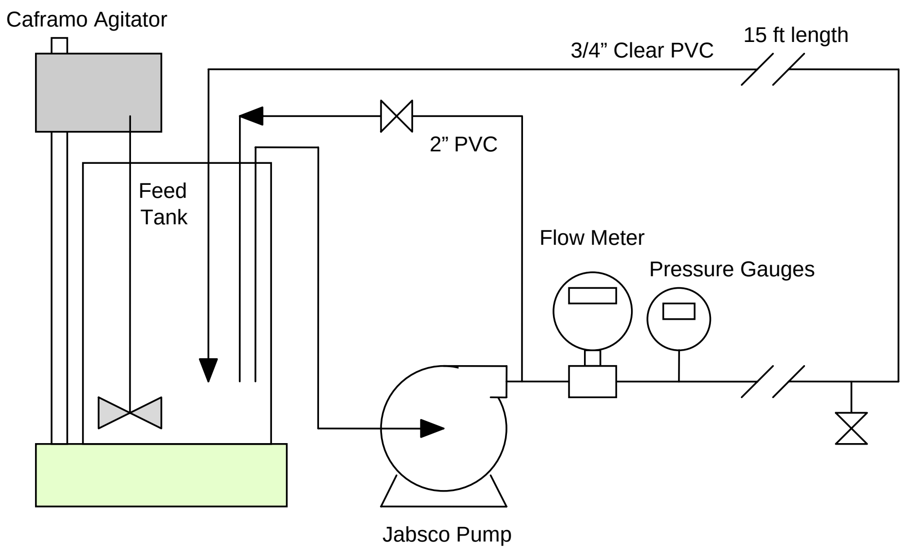

Figure 3. Schematic of Bead Pump Test Stand

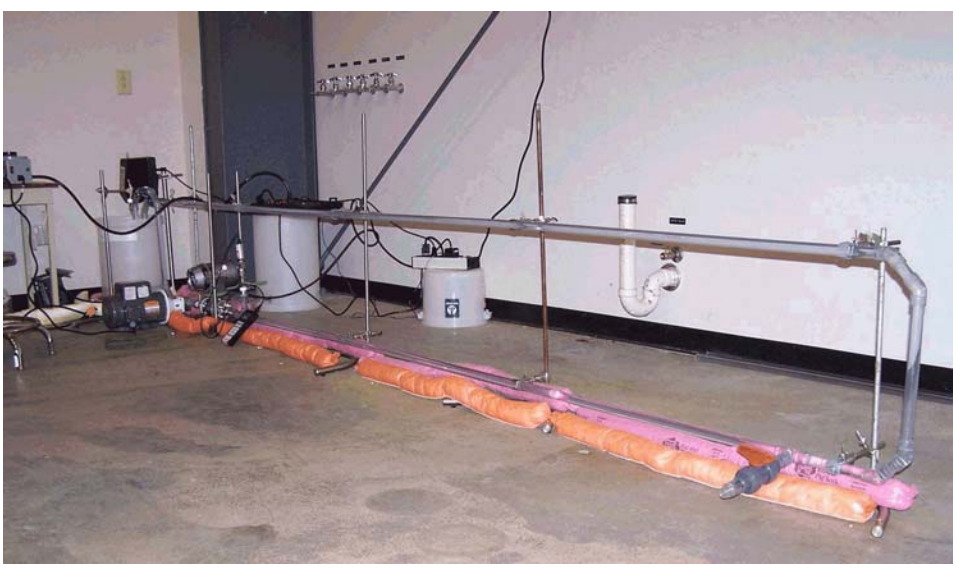

Figure 4. Bead Pump Test Stand

Overall, the data shows that the line pressure increased as the solids were increased for the same flow rate. In addition, the line pressure was higher for Frit

320 than the beads at the same solids level and flow. With the observations, a determination of minimum velocity to prevent settling could be done, but a graph of the line pressures (see Figure 5) versus velocity (not flow) for the various tests was deemed to more objective. The graph shows that the inflection point in pressure drop is about the same for the beads and Frit 320 (about 2.5 ft/sec). This indicates that the bead (-100+140 mesh) slurry would not require higher flows rates than frit slurry at DWPF during transfers. Another key finding was that the pump impeller was not significantly damaged by the bead slurry, while the Frit 320 slurry rapidly destroyed the impeller. Evidence of this was first observed when black particles were seen in the Frit 320 slurry being recirculated and then confirmed by a post-test inspection of the impeller. Finally, the pumping of bead slurry could be recovered even if flow is stopped. The Frit 320 slurry could not be restarted after stopping flow due to the nature of the frit to pack tightly when settled.

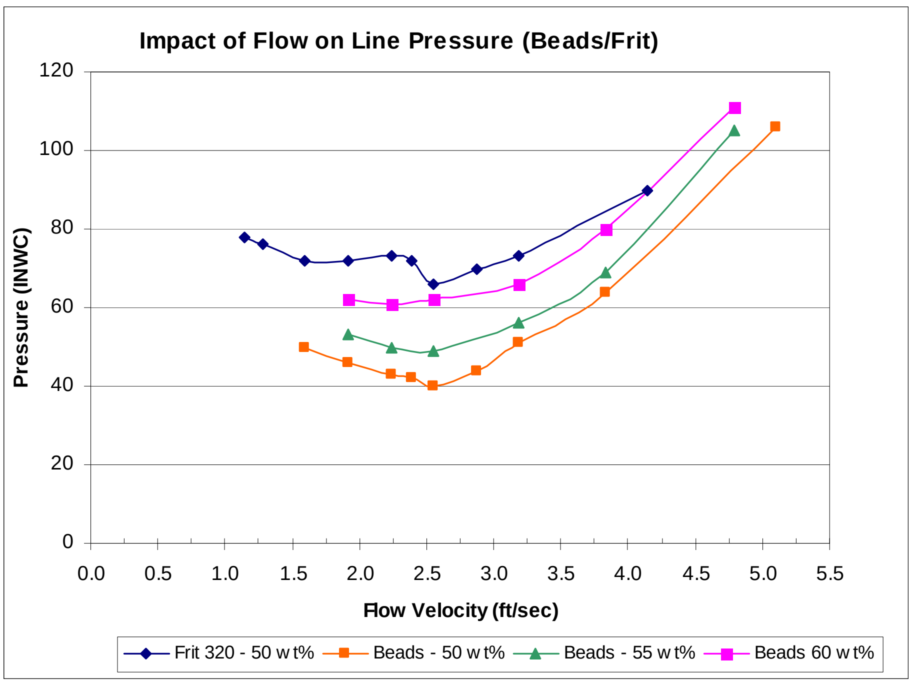

Figure 5. Line Pressure versus Velocity

Beads were shown to represent a significant process improvement versus frit for the DWPF process in lowering yield stress of the melter feed. Lower erosion of process equipment is another expected benefit.

Additional experimental work planned in the following areas:

- SME product homogeneity/sampling issues
- Melt rate during slurry-fed tests
- Impact of beads on frit addition equipment
    - Individual component evaluation
- Testing with various ratios of frit to beads

References

[1] Stone, M. E., Schumacher, R. F., 2004,"Preliminary Evaluation of Glass Former Morphology Impacts on Rheological Properties of Simulated DWPF Melter Feed Slurries", WSRC-TR-2004-00337, Washington Savannah River Company, Aiken, SC.

[2] Smith, M.E., Stone, M. E. Stone, D. H. Miller, 2005,"Impact of Spherical Frit Beads on Simulated DWPF Fluids" WSRC-TR-2005-00148, Washington Savannah River Company, Aiken, SC.